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DEVELOPMENT OF A MASS SPECTROMETER SYSTEM

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ABSTRACT

DEVELOPMENT OF A MASS SPECTROMETER SYSTEM - PHASE III

A cold cathode ion source (CCIS) of the magnetron type has been developed for use in ultrahigh vacuum residual gas analysis. The purpose is to eliminate the spurious spectra produced by the conventional, hot filament ion source and to produce increased sensitivity.

Studies were conducted using a commercial quadrupole spectrometer; the quadrupole is suitable because of its relative tolerance for a broad ion beam and a large spread in ion energies. The commercial unit was not optimally designed for the CCIS, but allowed both its performance and potential to be evaluated.

Cold cathode quadrupole performance was studied as a function of numerous parameters to determine optimum operating conditions. Resolution of mass peaks .7 amu apart was ultimately achieved, with the valley between peaks less than 10% the height of either adjacent peak. This resolution was identical to that obtained with the commercial hot filament source accompanying the spectrometer and appeared limited by the quadrupole itself. Sensitivity exceeded that obtained with the hot filament source.

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SUMMARY

A cold cathode ion source (CCIS) of the magnetron type has been developed for use in ultrahigh vacuum residual gas analysis. The purpose is to eliminate the spurious spectra produced by the conventional, hot filament ion source and to produce increased sensitivity.

Studies were conducted using a commercial quadrupole spectrometer; the quadrupole is suitable because of its relative tolerance for a broad ion beam and a large spread in ion energies. The commercial unit was not optimally designed for the CCIS, but allowed both its performance and potential to be evaluated.

Cold cathode quadrupole performance was studied as a function of numerous parameters to determine optimum operating conditions. Resolution of mass peaks .7 amu apart was ultimately achieved, with the valley between peaks less than 10% the height of either adjacent peak. This resolution was identical to that obtained with the commercial hot filament source accompanying the spectrometer and appeared limited by the quadrupole itself. Sensitivity exceeded that obtained with the hot filament source, which was run with a 3 ma emission current. Increased sensitivity factors of 1.5 to 4.5 were obtained depending on pressure and atomic mass; the corresponding absolute sensitivities at optimum resolution were .40 ma/Torr for N^{28} at 3×10^{-9} Torr and 1.2 ma/Torr for A^{40} at 1×10^{-7} Torr. Over the 10^{-8} and 10^{-9} Torr pressure (p) ranges investigated, quadrupole collector current (i_c) obeyed the relationship $i_c = kp^{1.2}$, where K is a constant; an exponent closer to unity is expected over a larger pressure range.

The above results were obtained utilizing a 2.2 KV anode potential accompanied by an ion retarding field between source and quadrupole; hollow cylindrical magnets provided 1220 gauss at the center of the source. Smaller magnets providing 840 gauss, and having a third of the volume of the larger magnets were also used; they gave comparable resolution but only half the sensitivity and they caused greater operating mode instability.

Following an investigation of source operating parameters, sensitivity and resolution were investigated as a function of quadrupole operating frequency and rf and dc operating potentials; this was to determine the extent of benefits obtainable with an improved quadrupole design. Results indicated a potential sensitivity improvement factor of 10 with the same source configuration; resolution would be equal to or better than that obtained thus far. If an improved quadrupole would allow enlarging the source exit apertures, a still greater fraction of the source's internal sensitivity, which is in the amps/Torr region, could be realized.

1. INTRODUCTION

Mass spectrometers commonly use a hot filament to supply electrons for molecular ionization. This method has a distinct advantage in that low energy ions are produced which are easily focused into a collimated beam for spectroscopic analysis. However, the hot filament causes certain gases to decompose and in ultrahigh vacuum analysis this confusion is compounded by evolution of gases from the source itself due to the heat generated. The purpose of work reported here, based on studies related in earlier reports,^{1,2} is to ascertain the mass spectrometry capabilities of a cold cathode ion source (CCIS), so that these difficulties can be eliminated.

The source is derived from the cold cathode magnetron total pressure gauge reported by Redhead³. This gauge was selected as a starting point because it is clean, it exceeds the sensitivity of the common hot filament gauge by a factor of 50, and it is useable down to the 10^{-14} Torr range. Compared to the hot filament source, the CCIS generates high ion energies. It has therefore been coupled to a quadrupole mass spectrometer where energy requirements are relatively non-stringent. The results of optimizing and characterizing the CCIS-quadrupole are discussed herein. Böhm and Günther^{4,5} also have reported results of combining a quadrupole analyzer with a cold cathode ion source, of the Penning type. This combination is found useful for pressures above about 10^{-6} Torr, but the source was not operated in the ultrahigh vacuum region.

2. APPARATUS

2.1 Vacuum System

The vacuum system used is shown schematically in Figure 1. It is divided into upper and lower manifold units, which allows the upper portion to be oven baked while being pumped from below. Subsequent to bakeout, the upper manifold, thus cleaned, is valved off and pumped by the upper ion pump.

The upper manifold consists of the quadrupole spectrometer, a Modulated Bayard-Alpert gauge, an 11 liter per second Ultek Model 10-252 ion pump, a bakeable Varian ultrahigh vacuum valve for isolating the upper manifold from the lower and a bakeable Varian valve for isolating the upper ion pump. A thermostatically controlled oven is used for baking the upper manifold system. Figures 2 and 3 show two views of the experimental apparatus.

The lower manifold consists of a 20 liter/second Ultek Model 10-354 ion pump, a 2400 liter/second mechanical pump, a Varian zeolite sorption pump, a port for feeding gas to the system and associated valves for closing off the pumps and gas supply inlet. The pumping speed of the system was intentionally made small to detect outgassing in the CCIS spectrometer.

2.2 Quadrupole Spectrometer

On the basis of a feasibility study of the cold cathode ion source⁶, it was concluded that a quadrupole mass spectrometer would be most suitable for analysis of the source's higher ion energies and larger beam cross section. An Ultek Model 200 quadrupole was selected, equipped with a standard hot filament ionizer. The latter permitted spectrometer performance comparisons between both types of sources. A discussion of the comparative characteristics of three commercial quadrupoles and of the reasons for selecting the Ultek unit is given in Appendix A.

None of the commercially available analyzers had the optimum characteristics desired for operation with the CCIS for reasons to be discussed in Appendix A. However, the Ultek unit was chosen because it had the best resolution available and because it was capable of scanning automatically in a mode in which peak width remains constant with mass; this is desirable for the CCIS because ion energy restrictions are less stringent and because sensitivity vs. ion energy is constant with mass. The spectrometer was used

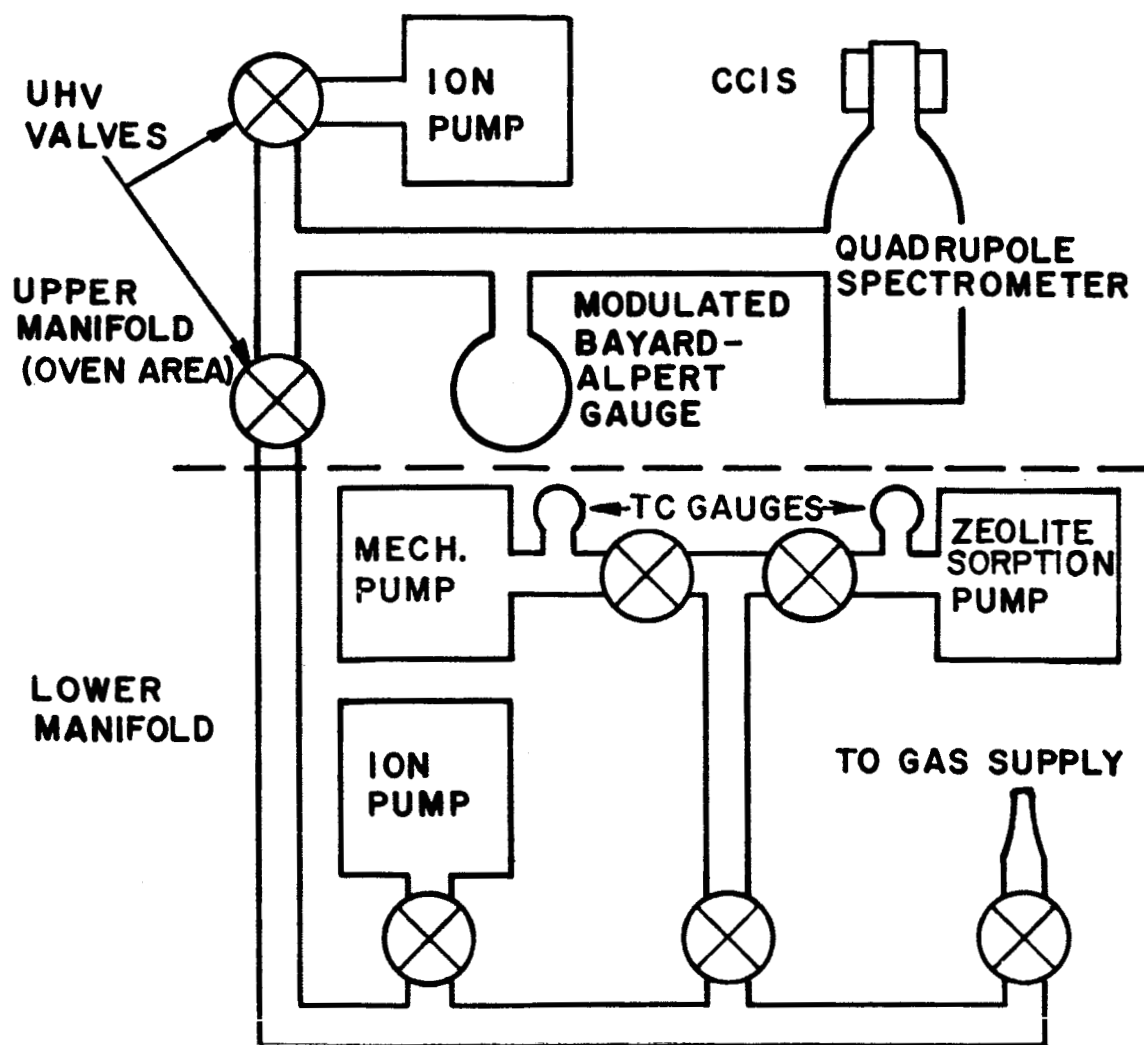


Figure 1
Vacuum System Schematic

CC12\0nsqubofe-snn nna gasetw ya vllsuged tot Ctenutness tests

Figure 3

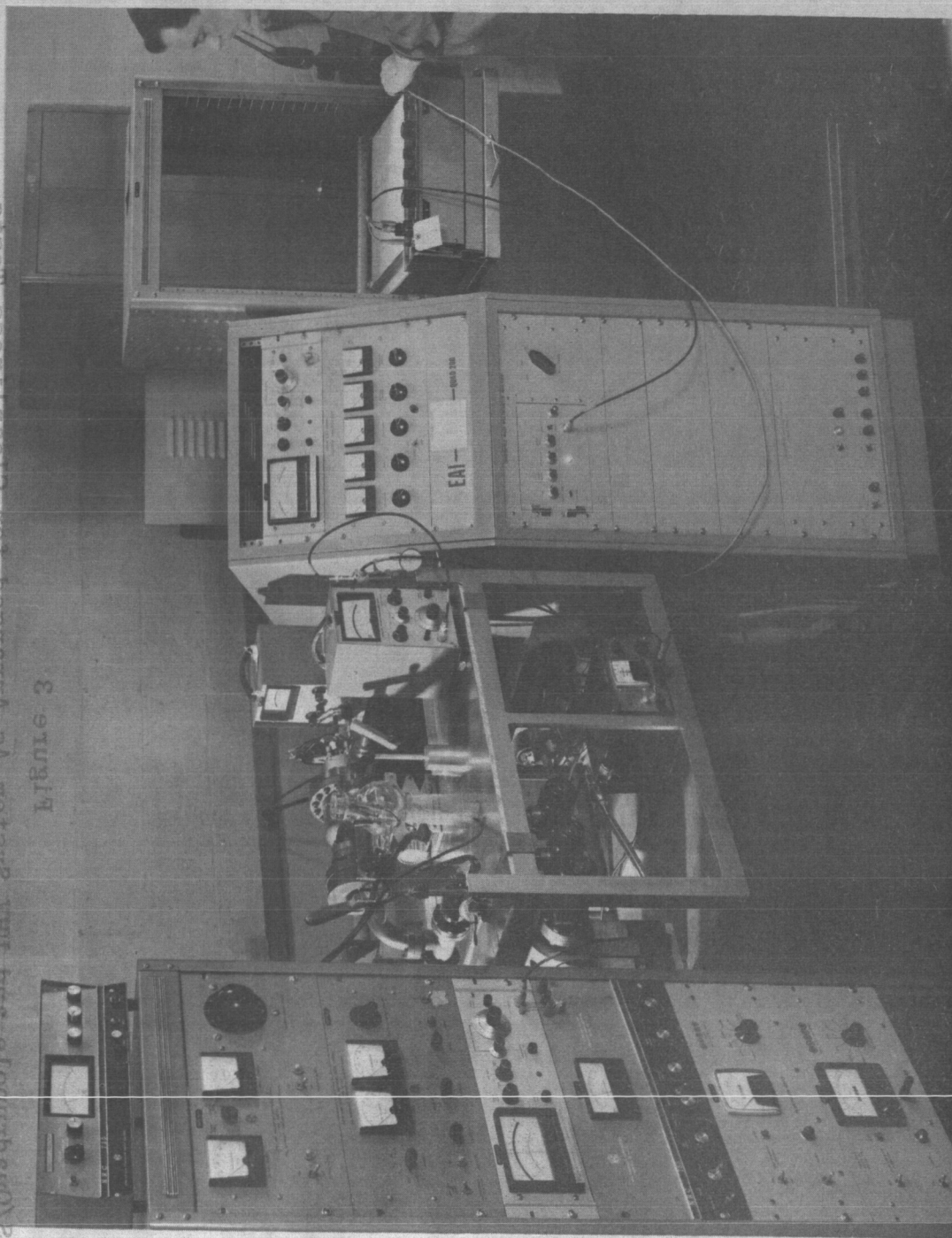


Figure 2
Experimental Test Apparatus

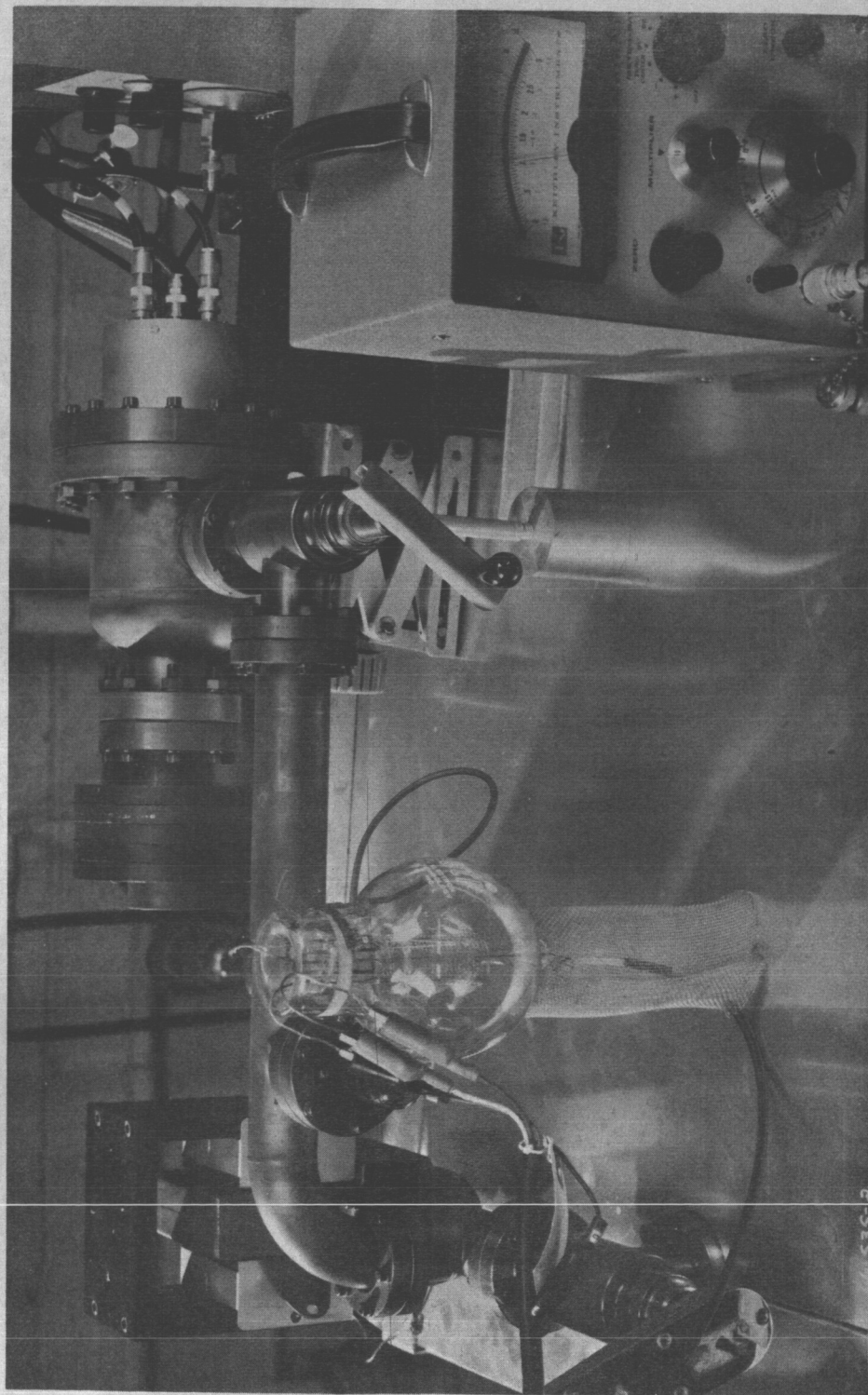


Figure 3
CCIS/Quadrupole and UHV System As Arranged for Cleanliness Tests

with a Faraday cup ion collector rather than with an electron multiplier to allow determination of basic spectrometer characteristics, such as sensitivity, without amplification variations and with a minimum of spectral perturbations.

Quadrupole specifications pertinent to a theoretical analysis of results are as follows: Operating frequencies (ν) are 5.3 mc and 3.3 mc on the low and medium mass ranges, respectively. Maximum available rf potential (V) is 1000 volts. The radius (r_0) of the circle inscribed by the quadrupole rods is 2.7 mm. The entrance aperture diameter is also 2.7 mm. Rod lengths are 11.3 cm.

2.3 Cold Cathode Ion Source

The cold cathode ion source (CCIS) is a modification of the Redhead magnetron gauge. An assembly drawing of the source attached to the quadrupole is shown in Figure 4. To allow ion extraction, the single spool shaped cathode design used in the gauge is separated into two cathodes, one with a remnant stub (K_1) which is 9.7 mm long and 3.1 mm in diameter. All remaining electrode dimensions remain as in the gauge. The horseshoe magnet ordinarily used has been replaced by hollow cylindrical ceramic magnets. Dimensions and field strengths of the magnets used are given in Section 3.7. Figure 5 shows two views of the CCIS/Quadrupole without its external housing.

Source electrodes are supported on a stainless steel cylindrical housing maintained at ground potential. Auxiliary cathodes are welded to it directly. The anode is attached and insulated by sapphire spheres. It is not perforated as in the Redhead gauge. Cathodes K_1 and K_2 are supported and insulated by glass studs. This arrangement provides independent electronic access to the anode and each cathode; it also provides a guard ring design which, by preventing leakage between electrodes, prevents interference with low level current detection.

The source housing is perforated and attached to the quadrupole with heavy refractory wire and a slotted sleeve; distance between the faces of K_2 and the quadrupole aperture plate was maintained at 2.4 mm (as close as possible without causing arcing between electrodes). Rigidity and higher conductance to the exhaust port were thus provided.

Tungsten mesh screens were used over the K_2 aperture and the quadrupole entrance aperture for the following reasons: (a) to allow testing of the effect of potential differences between these components without causing beam defocusing, (b) to isolate magnetron and quadrupole electric fields and (c) to flatten the magnetron potential distribution at K_2 which otherwise bulges through the K_2 aperture; this is to reduce ion defocusing and resultant sensitivity losses.

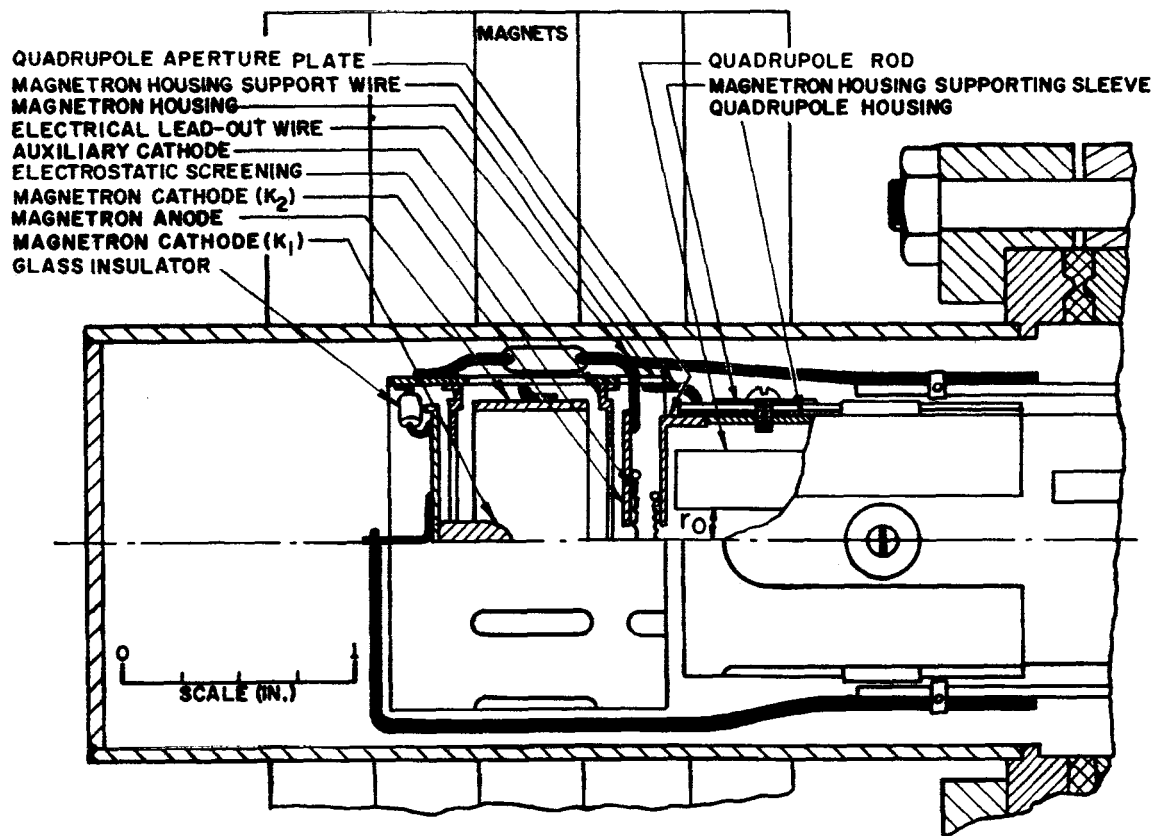
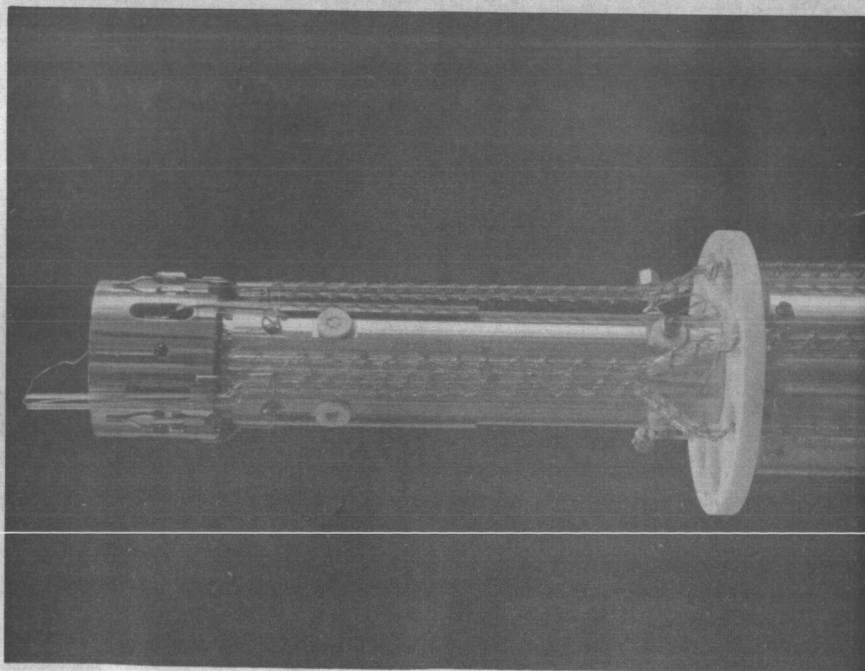
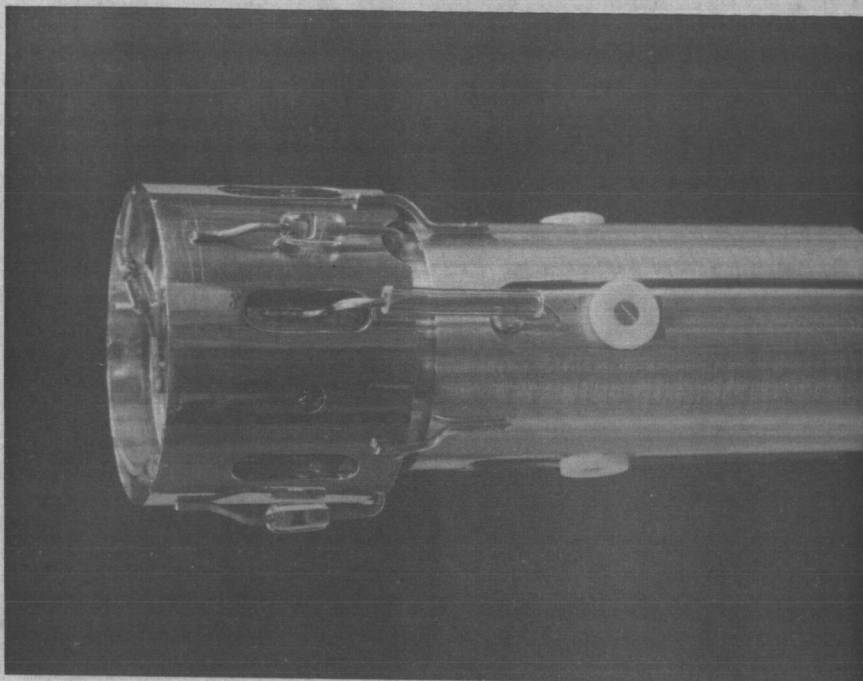


Figure 4

Cross-Section of Cold Cathode Ion Source
Showing Attachment To Quadrupole Analyzer



(a) Overall View



(b) Close-up of CCIS

Figure 5
Two Views of CCIS/Quadrupole

3. PROCEDURES AND RESULTS

Quadrupole spectrometer performance was first investigated with the manufacturer's hot filament source, which accompanied the quadrupole. This was to achieve familiarity with the spectrometer, to determine its characteristics and to enable performance comparisons to be made between the two sources. Studies utilizing the hot filament source are discussed in Appendix B.

CCIS/Quadrupole performance was investigated as a function of the parameters discussed below, first to determine optimum source operating conditions and second, to note the resultant spectrometer response to mass and pressure variations. During the first part of the investigation, nitrogen was used as the test gas at a pressure of 5.4×10^{-9} Torr. Initially, a 5.0 KV anode potential was used and cathode potentials V_{K1} and V_{K2} were kept at zero volts; thereafter, values were used which successively were shown to give the best resolution and sensitivity.

3.1 Performance vs. Quadrupole Resolution Setting

A family of nitrogen spectra were recorded as a function of quadrupole resolution dial settings. The latter correspond to ratios of dc and rf rod potentials, U and V respectively, in Paul's⁸ equation for quadrupole spectrometer resolution

$$\frac{M}{\Delta M} = \frac{0.126}{0.16784 - U/V} \quad (3.1.1)$$

where M is the atomic mass and ΔM the peak width at its base. Spectral resolution changes were determined by comparing the full width at half maximum (FWHM) of the nitrogen peaks. Experimental resolution improved as dial settings proceeded toward higher theoretical resolution; sensitivity correspondingly decreased (as predicted by theory) until a setting was reached (4.95) beyond which sensitivity continued to decrease with no further resolution improvement. This setting was then selected as optimum and used in subsequent experiments.

3.2 Performance vs. CCIS Anode Potential

As with resolution setting experiments, FWHM measurements were made vs. anode potential (V_A) on a family of nitrogen spectra. This was done both on the low and medium mass ranges (1-50 amu and 10-150 amu, respectively). The optimum value of V_A for both ranges was in the vicinity of 2.0 KV (compared with 5.0 KV normally used in magnetron total pressure gauges). Figure 6 illustrates, for the medium mass range, the resolution improvement and accompanying sensitivity reduction existent as a function of V_A . (It is evident that the discharge was unstable at 5.0 KV).

3.3 Performance vs. CCIS Magnet Position

Using the optimum resolution setting and optimum anode potential discussed above, spectra were recorded against magnet position; first axially and then rotationally. Positions giving optimum resolution, as determined by FWHM, were selected. Unlike spectral variations with resolution setting and with anode potential, no sensitivity loss accompanied the positionally improved resolution. Resolution was found optimum when the magnet's center coincided with the midplane between cathodes K_1 and K_2 . A + 3 mm axial magnet movement caused about a 20% broadening of the peak's FWHM. A 10% FWHM maximum variation occurred with rotational positioning.

3.4 Performance vs. CCIS Ion Retarding Potential

Acceleration and retardation of ions prior to their entrance to the quadrupole was effected by equal alteration of cathode potentials V_{K_1} and V_{K_2} while the quadrupole entrance aperture was maintained grounded. Acceleration was found to degrade quadrupole resolution while retardation improved it. This improvement, coupled with that obtained by lowering the anode potential (discussed above), points to high ion energy as an important resolution limiting factor.

Nitrogen peak narrowing, produced by increasing retardation potentials, is shown in Figure 7 for some of the values investigated. A 300 volt retardation potential was found optimum, when accompanying a 2.0 KV (later 2.2 KV) anode potential. For this determination the 10% valley criterion* was used, the FWHM criterion being abandoned due to improved resolution.

*Peak separation which allows a valley less than 10% the height of either adjacent peak. With one peak, an equivalent criterion was used; the separation between 5% of the peak height on either side of the peak's center line.

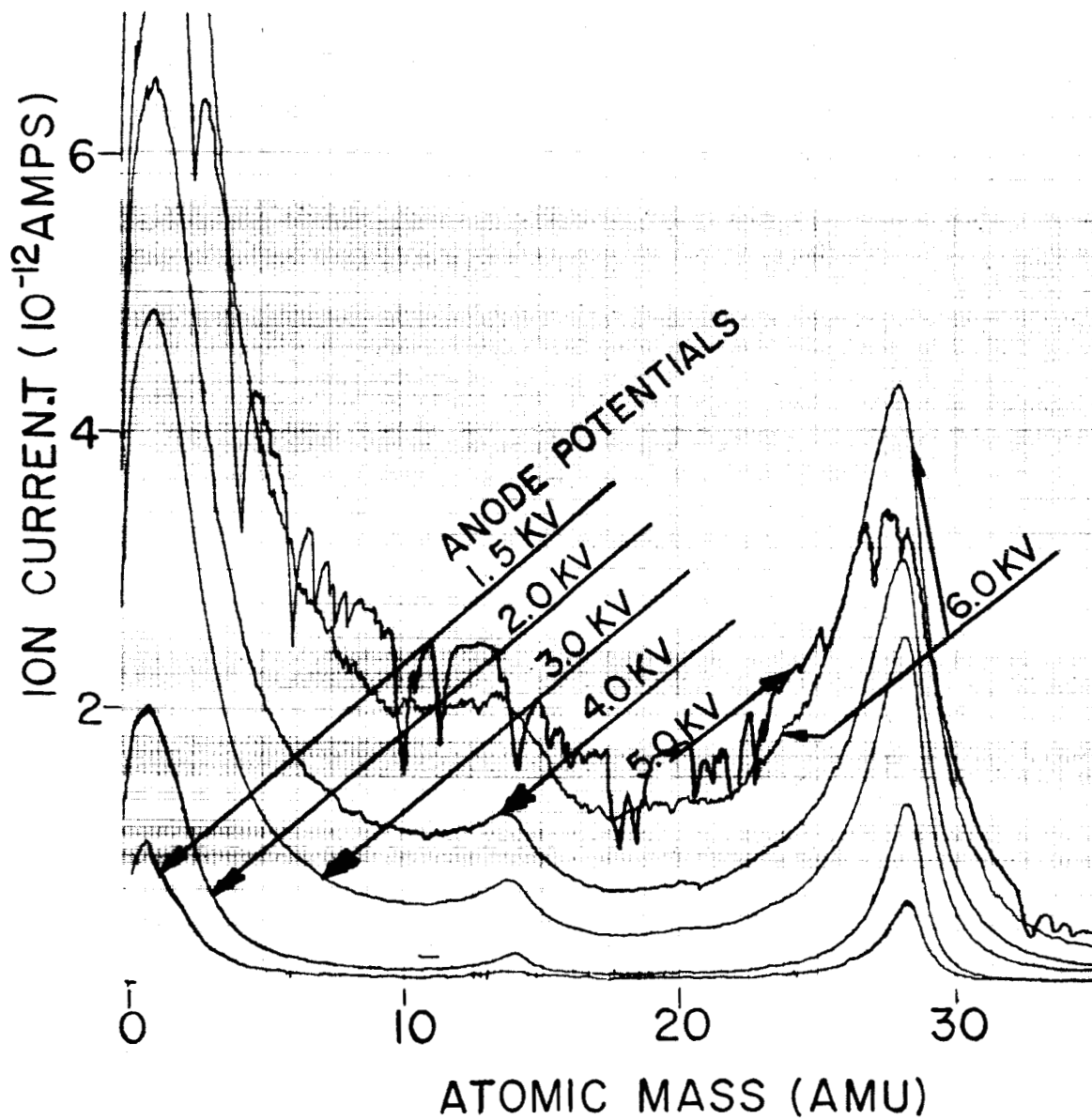


Figure 6

Resolution and Sensitivity Vs. Anode
Potential-Medium Mass Range

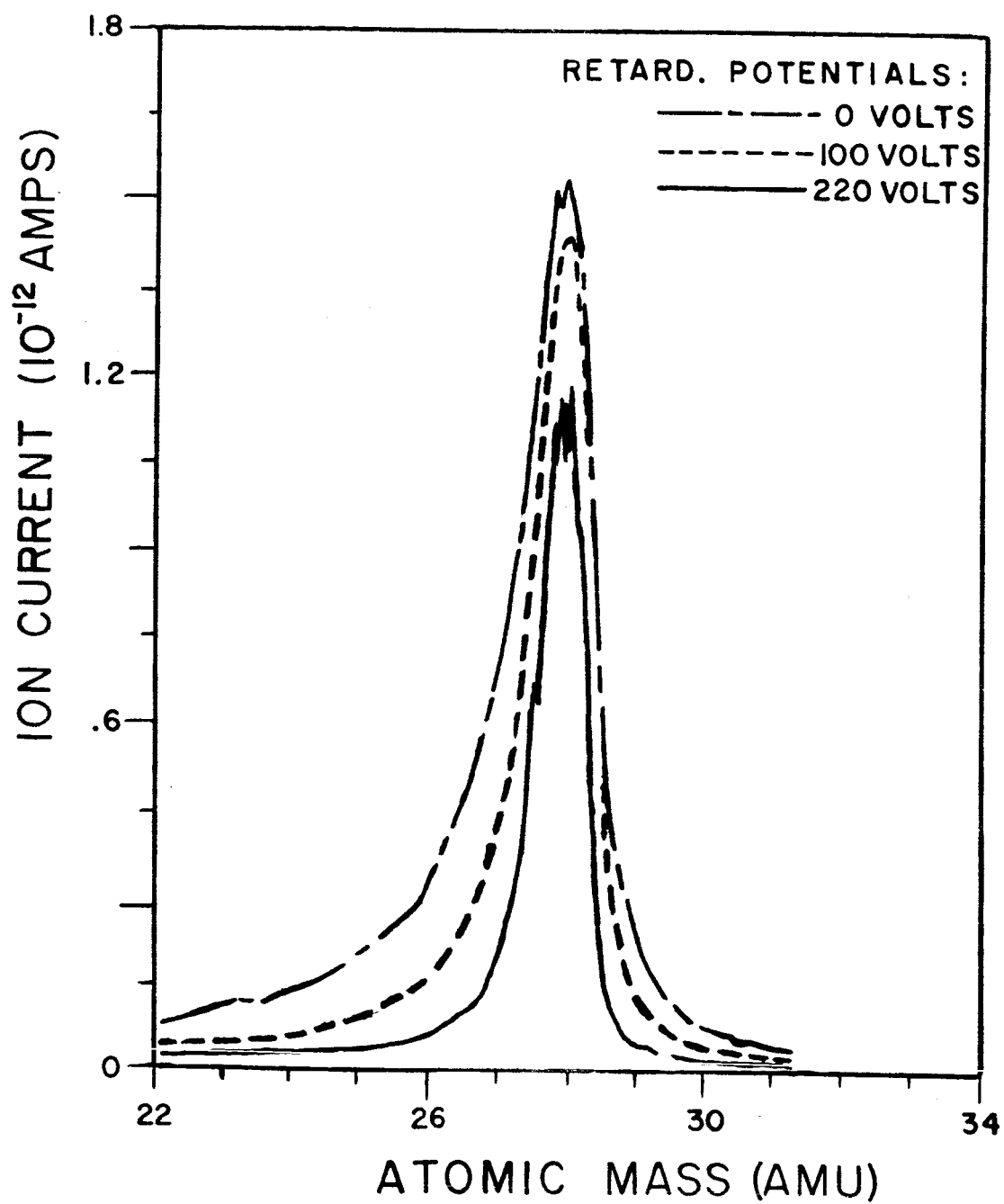


Figure 7
Effect of Retarding Potential On Resolution and Sensitivity
of Nitrogen Peak

3.5 Performance vs. Potential Between CCIS Cathodes

When making cathode potential $V_{K_1} > V_{K_2}$, resolution deteriorated obviously. For $V_{K_1} < V_{K_2}$, two conditions were tested: (1) with V_{K_2} at -300 volts, resolution remained constant and sensitivity declined, (2) with V_{K_2} at zero volts, resolution improved but not to the extent achieved by equal variation of cathode potentials, noted in Section 3.4 above. This investigation was, therefore, concluded.

3.6 Performance vs. Quadrupole Mass Range (Frequency)

Figure 8 shows nitrogen spectra obtained on the low and medium mass ranges using optimum conditions, as discussed above. Only a portion of the spectra are shown and scales are normalized to the same mass scale. The superior resolution and sensitivity of the low mass range is obvious. (The noise on the medium mass spectrum is in the X-Y recorder.) Resolution at this stage of investigation (using the 10% valley criterion) allowed peak separations of 2.5 amu and 1.5 amu for the medium and low mass ranges, respectively. These values were reproducible. The implications of the spectral differences as a function of mass scanning range will be discussed in Section 4.

3.7 Performance vs. Magnet Size

Experiments heretofore described, were performed with a hollow, cylindrical, ceramic magnet stack with a 11.4 cm O.D., a 4.8 cm I.D. and a 5.7 cm length. Most of these experiments were repeated using a smaller magnet stack with a 8.9 cm O.D., 4.8 cm I.D. and 3.8 cm length. Axial magnetic flux densities, in gauss, for the large and small magnets, respectively, were as follows: (a) on axis at the magnet's center, 1220 and 838; (b) on axis at the magnet's edge, 397 and 368; (c) on the inside wall of the magnet at the magnet's center, 1426 and 957.

The spectra obtained with the small magnet were very similar to those obtained with the large magnet. Resolution at this stage of investigation, allowed peak separations of 3.0 amu and 1.5 amu (using the 10% valley criterion) on the medium and low mass ranges, respectively. This compares with 2.5 amu and 1.5 amu, respectively, for the large magnet. Sensitivity was slightly better than half that obtained with the large magnet. Most significantly, the small magnet displayed comparatively large operating mode instabilities; during one mode change, the entire nitrogen peak temporarily disappeared.

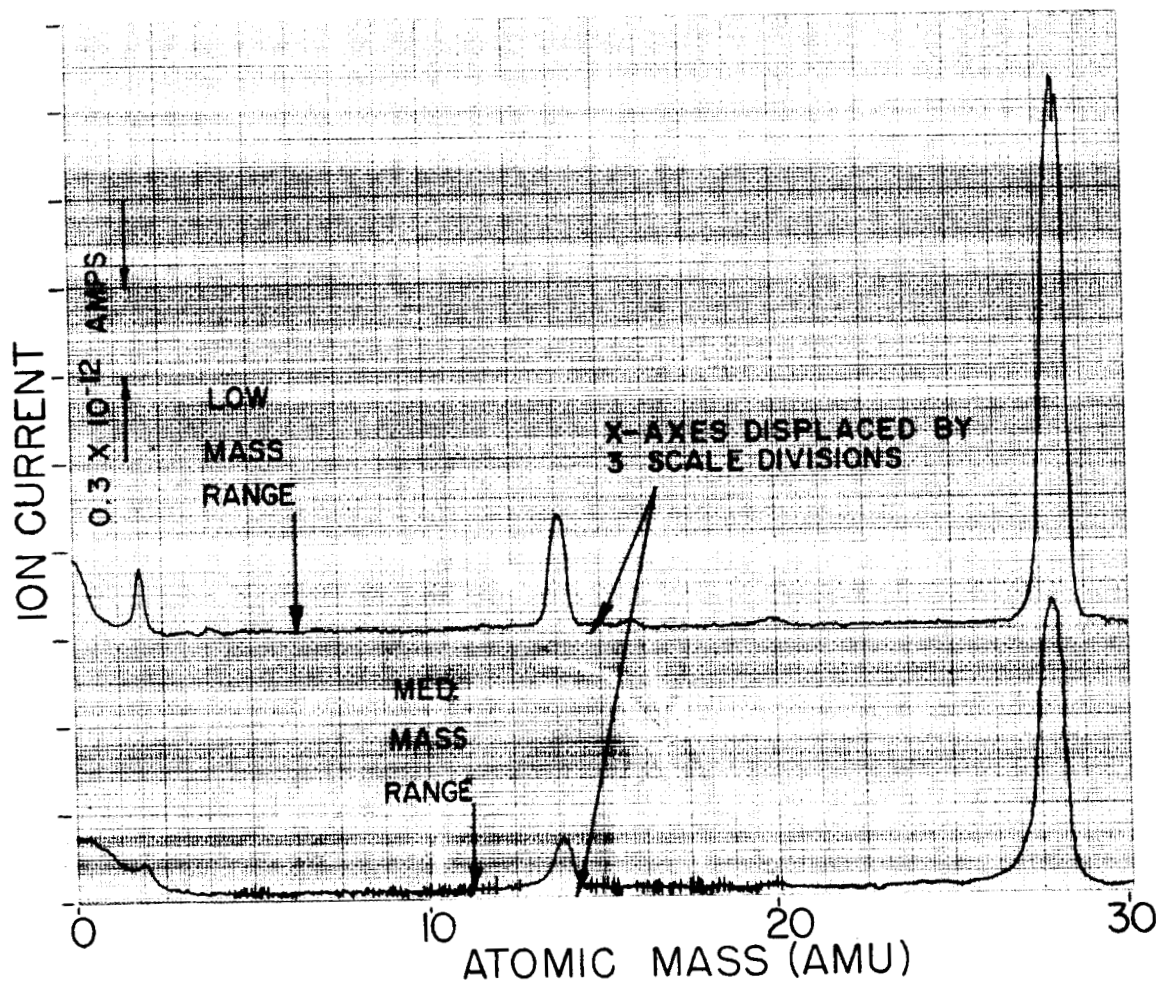


Figure 8
Performance of CCIS/Quadrupole Spectrometer
on Two Mass Ranges

Further development might establish reliable operation with little or no loss of resolution or sensitivity, perhaps for still smaller magnets. At present, however, the larger magnet gives a more stable and sensitive performance.

3.8 Performance vs. Spectrometer Cleanliness

The CCIS/Quadrupole and upper manifold were baked at 425°C for two days prior to the tests to be described next. This was in preparation for testing collector current linearity with pressure and for determining absolute sensitivity.

The baking resulted in an unexpected resolution improvement. Peak separations of 1 amu and .7 amu in the medium and low mass ranges, respectively, were obtained where 2.5 amu and 1.5 amu existed previously. The poorer resolution in the dirtier system is believed produced by increased secondary ion emission, caused by ions or electrons from the source bombarding the quadrupole rods. Secondary ions thus produced part way down the rods would not be fully resolved and could cause the observed resolution deterioration.

As noted in the summary, one of the objectives of this program is to improve the cleanliness of a quadrupole spectrometer by using a cold cathode ion source. Accordingly, certain tests were conducted during the program in an attempt to determine, at least qualitatively, the cleanliness of the spectrometer. It was not possible, however, to compare the hot cathode and cold cathode versions of this spectrometer simultaneously under the same conditions. Therefore, the results to be described next must not be considered completely conclusive.

To simulate a hot filament ion source, the Modulated Bayard-Alpert gauge was used. When the gauge is operating the processes which give rise to the spurious spectra (C^+ , O^+ , CO^+ , CO_2^+ , etc) in hot filament spectrometers, should also occur in the Bayard-Alpert gauge. The magnitude of these anomalous peaks will depend on many factors such as filament temperatures, electron emission and on initial gauge cleanliness. Therefore, the Bayard-Alpert gauge operation should give rise to the production of these spurious peaks. It is to be noted, however, that the Bayard-Alpert gauge has had the benefit of a rigorous outgassing procedure and the entire system had been pumped down for approximately 30 days before the first test to be described was begun. Figure 9 shows the results of this test. The test sequence is from the bottom to the top of the figure and the scans are labelled I through V. Scan I shows the background spectra of the system as determined by the CCIS/Quadrupole. The Bayard-Alpert gauge has been turned off for 18 hours.

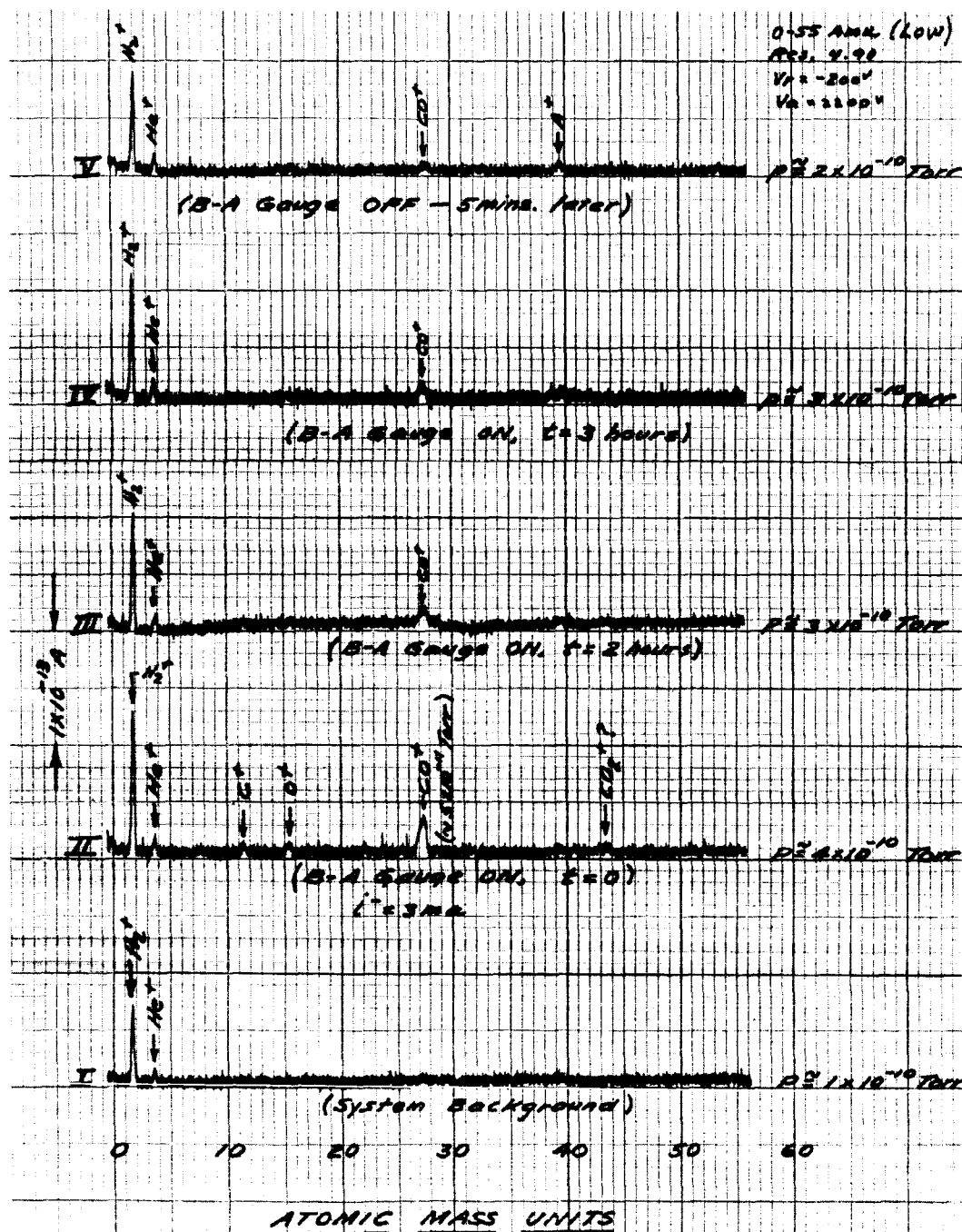


Figure 9
 Background Spectra Produced by Bayard-Alpert Gauge

Scan II shows the spectrum immediately after turn-on of the Bayard-Alpert gauge. The typical peaks of C^+ , O^+ , CO^+ , and possibly CO_2^+ are seen. Scans III and IV show the CO^+ peak 2 and 3 hours after initial start of the Bayard-Alpert gauge. It is noteworthy that the CO^+ peak is still present. Scan V shows conditions only five minutes after turning off the Bayard-Alpert gauge. The CO^+ peak has decayed more noticeably in the five minute period between IV and V than in the one hour period between scans III and IV. It is obvious that the initiation of the hot-filament gave rise to certain peaks which are not easily removed, despite continuous pumping, as long as the Bayard-Alpert gauge is operating. The production of these peaks therefore is not simply a single desorption process, but involves a desorption-adsorption cycle requiring a long time to overcome. A number of such cyclic interchanges are known to exist within Bayard-Alpert gauges.

The H_2^+ and He^+ peaks are also noticeable in all the scans. The hydrogen peak (H_2^+) will be discussed shortly. The helium (He^+) peak is probably a remnant from an accidental over-exposure to helium (above 10^{-3} Torr) which occurred weeks before. The system was not baked subsequent to this exposure.

Figure 10 shows a second attempt at defining spectrometer cleanliness. For this test, the UHV valve shown in Figure 1 between the upper ion pump and upper manifold was removed and placed between the CCIS/Quadrupole spectrometer and the upper manifold. This configuration is shown in Figure 3. With this valve intervening the spectrometer and the system, it is possible to isolate the spectrometer. Three conditions are shown in this figure. In the first, the spectrum of the entire system is shown. The upper ion pump and Bayard-Alpert gauge are operating and the valve between the spectrometer and the system is open. The middle scan was taken with the ion pump off to determine what gases are being removed by the ion pump. In both the foregoing cases, hydrogen (H_2^+) is the only residual detectable. When the spectrometer is isolated we notice a very large increase in the H_2^+ peak, together with a small Helium (He^+) peak. The source of the hydrogen peak is not known. It may be anywhere inside the spectrometer and/or valve configuration. Present speculation is that the stainless steel of the spectrometer housing may be the source. Redhead* has experienced similar results using a rigorously cleaned hot-filament spectrometer attached to a stainless steel system. In any event,

* From a private communication

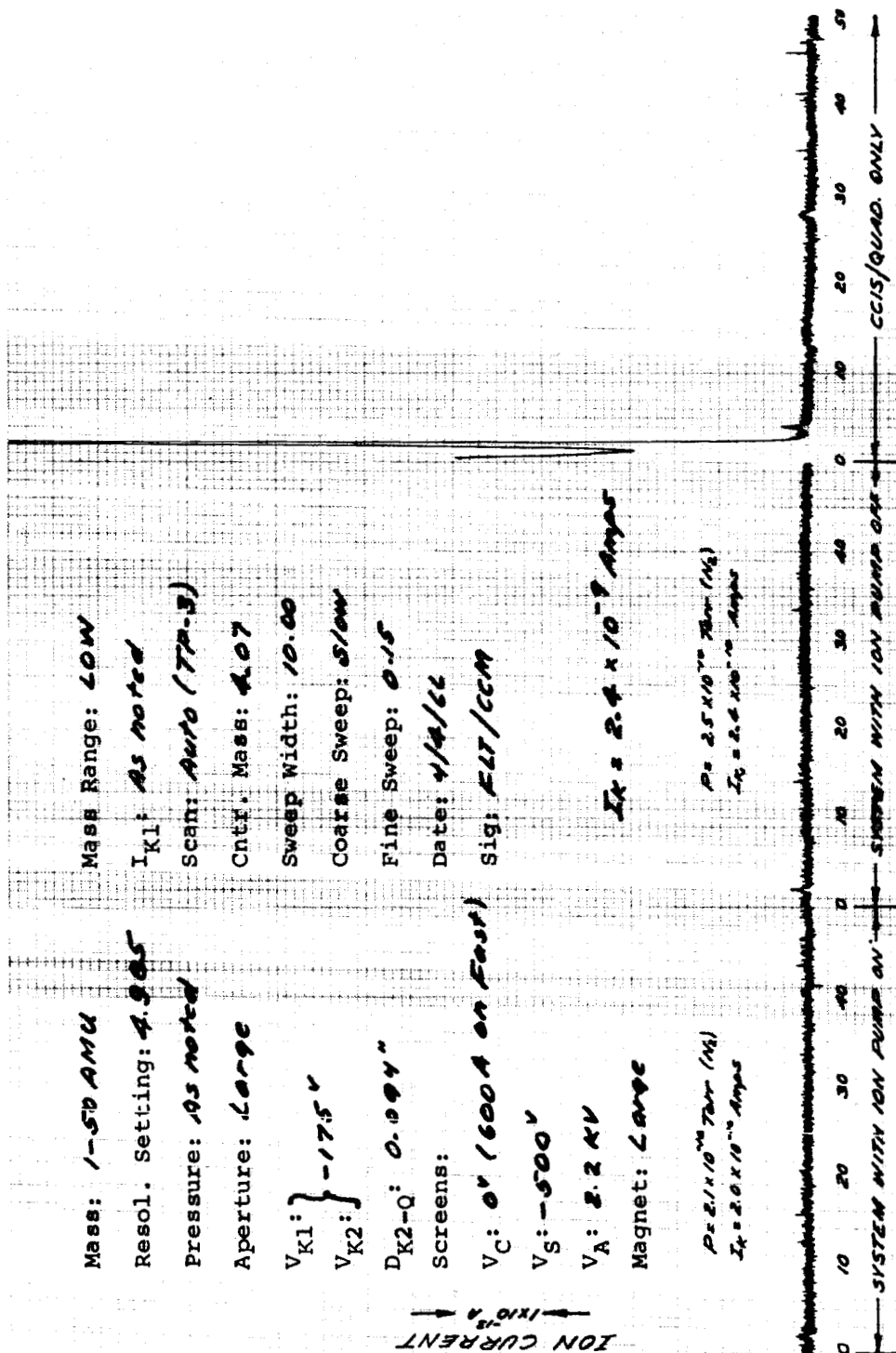


Figure 10
Background Spectra From CCIS/Quadrupole

the familiar C^+ , O^+ , CO^+ and CO_2^+ spectra were not observed with the spectrometer isolated. The center scan (ion pump off) raises the question as to why so little hydrogen was observed with the ion pump on or off. The answer is that the clean titanium surfaces in the ion pump is an excellent hydrogen getter whether the pump is operating or not.

As noted previously, and as will be noted in a later section, conclusive tests of the CCIS/Quadrupole cleanliness must await a simultaneous comparison between the hot and cold cathode versions of the instrument under similar conditions. With the exception of hydrogen, only remnant amounts of residual gases have been noted.

3.9 Performance vs. Pressure

3.9.1 Resolution vs. Pressure - Resolution improved slightly with increased pressure, but ion retarding potential had to be reduced to prevent spectral extinction; ion energy apparently decreases with increasing pressure. In accordance, a retarding potential just under that causing spectral extinction appeared optimum; the values noted at several pressures were as follows: 300 volts at 5.4×10^{-9} Torr, 200 volts at 3.8×10^{-8} Torr, 75 volts at 1.2×10^{-7} Torr, 54 volts at 2.3×10^{-6} Torr.

Figure 11 shows a medium mass range spectrum of an inert gas mixture, obtained at 2.3×10^{-6} Torr subsequent to a $425^\circ C$ bakeout. It is evident from the Xe^{131} and Xe^{132} peaks that a 1 amu separation has been obtained using the 10% valley criterion. On the low mass range resolution better than .7 amu was obtained. A low mass range methane spectrum at 2.5×10^{-8} Torr is shown in Figure 12; although not optimum, the same retarding potential was used as at 2.3×10^{-6} Torr. A .7 amu peak separation can be deduced at mass 16 using the single peak 10% valley criterion. This resolution is equal to or better than that obtained by the authors using the hot filament source supplied as standard equipment with this quadrupole; it appears therefore that resolution, at this point, is limited by the quadrupole itself.

It is of interest to note the very small peak at mass 17 which is fully resolved, although only one mass unit separates it from the large mass 16 (CH_4^+) peak. Therefore, resolution is greater on the higher mass side of a given peak than on the lower side.

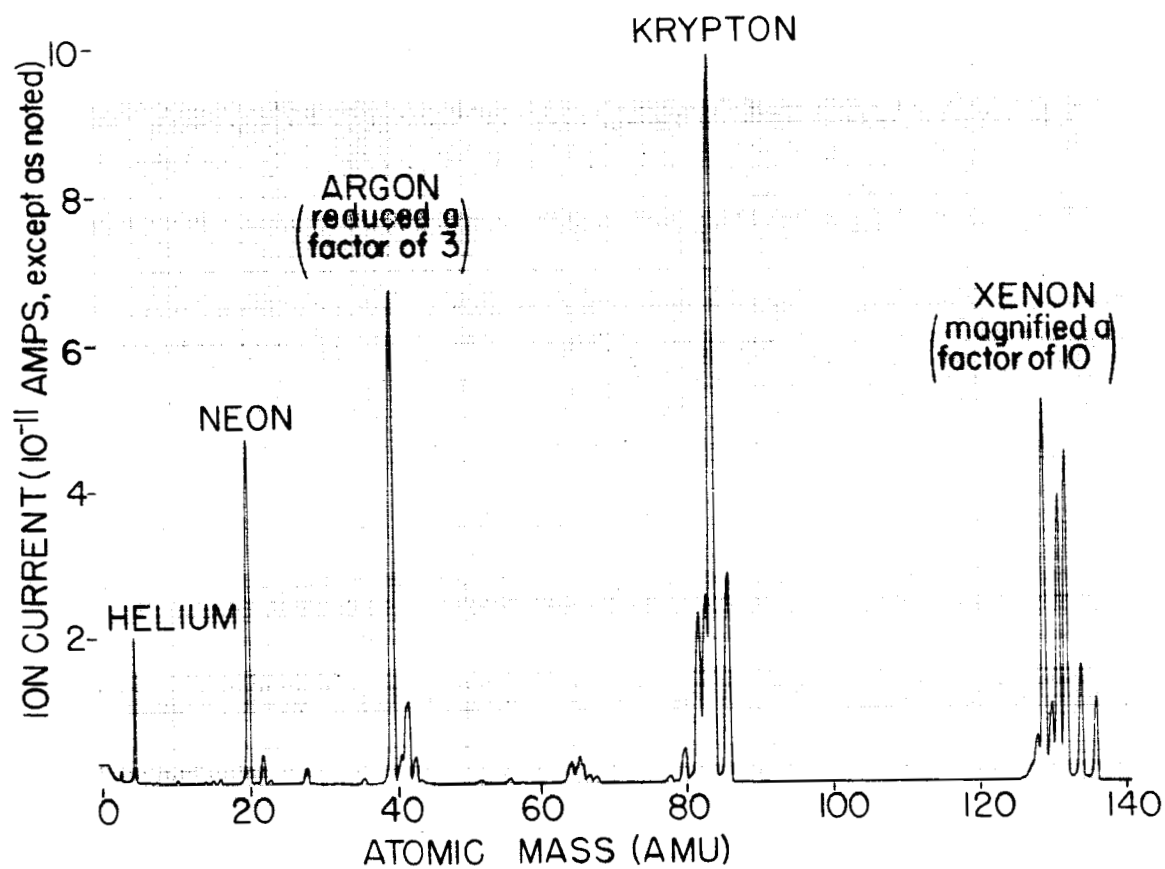


Figure 11
Performance of CCIS/Quadrupole on Medium Mass Range
For Inert Gas Mixture

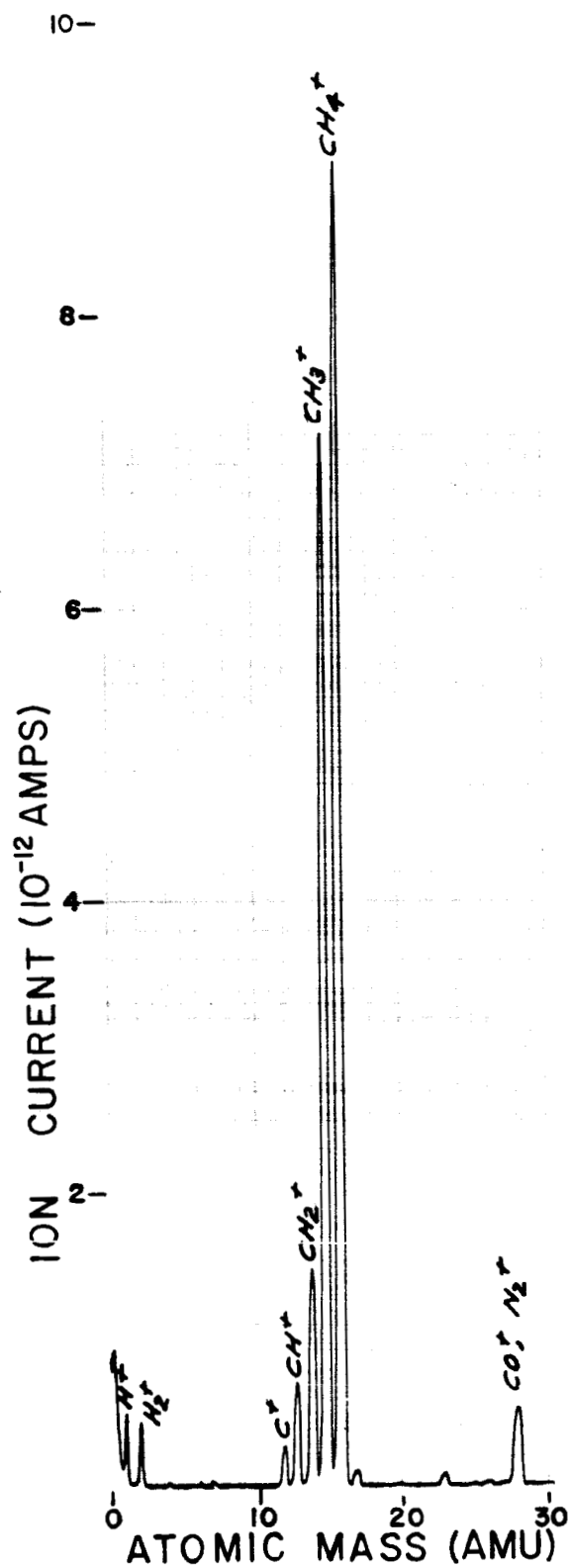


Figure 12

Performance of CCIS/Quadrupole on Low Mass Range-Methane Spectra

collector was investigated as a function of pressure from approximately 3×10^{-9} Torr to 1×10^{-7} Torr for He^4 , Ne^{20} , N^{28} and A^{40} on the low mass range and for A^{40} and Kr^{84} on the medium mass range. A constant 100 volt ion retarding potential was used and the following procedure adopted: (a) Bayard-Alpert gauge emission current was adjusted for each gas, according to relative sensitivities tabulated for Dushman and Young in Dushman⁹, to give a constant pressure sensitivity of .01 amps/Torr, (b) The quadrupole was set manually at the peak of the mass under investigation, (c) The ion pumps were valved off and the gas under investigation admitted slowly to maintain pressure equilibrium between gauge and ion source, (d) Quadrupole collector current was then continuously plotted against Modulated Bayard-Alpert gauge current.

Ion current at the quadrupole collector (i_c) vs. pressure (p) was found to obey the relationship

$$i_c = kp^n \quad (3.9.3)$$

(where k is a constant) for N^{28} , and Kr^{84} , while He^4 and Ne^{20} obeyed equation 3.9.3 only in the 10^{-8} Torr range. A decrease in the exponent occurred for the latter gases with decreasing pressure in the 10^{-9} Torr range and was believed due to a leak in the system which was discovered after the data was taken; the low ionization efficiency of He^4 and Ne^{20} makes them more susceptible to these problems. The exponent n was between 1.15 and 1.20 for nitrogen, argon, and krypton; for neon and helium, it was .927 and .950, respectively.

A more thorough investigation of ion current over a wider pressure range is necessary to establish the value more precisely; this is due to the oscillations and discontinuities existent in cold cathode discharges over wide pressure ranges. The exponent may then more closely approximate unity, as it does in the magnetron gauge.

3.10 Performance vs. Atomic Mass

3.10.1 Sensitivity vs. Atomic Mass - Sensitivity vs. atomic mass for the gases investigated is shown in TABLE I. Values are tabulated at 1×10^{-7} Torr and 3×10^{-9} Torr, which are the mean pressure extremes investigated. Sensitivities are derived from representative curves drawn from the data, which follow the relationship given by equation 3.9.3.

The lower pressure values for helium and neon are extrapolated due to leak difficulties discussed in Section 3.9.2. Sensitivities obtained at 1×10^{-7} Torr with the hot filament source operated at a 3 ma emission current is also tabulated for comparison; this source was a standard accessory of the spectrometer. Resolution for both hot filament and cold cathode sources was approximately 1 amu for all values shown. Sensitivities have been corrected for relative atomic abundances and for Bayard-Alpert gauge sensitivity variation with gas composition. The tabulated sensitivities were obtained with a Faraday cup collector and are therefore unamplified.

TABLE I

Spectrometer sensitivity vs. atomic mass for hot filament and cold cathode ion sources. L and M indicate low and medium mass range results, respectively.

Gas	Sensitivity (ma/Torr)		
	CCIS		Hot Fil.
	3×10^{-9} Torr	1×10^{-7} Torr	1×10^{-7} Torr
He ⁴	.077 ^a (L)	.064 (L)	.042 (L)
Ne ²⁰	.13 ^a (L)	.10 (L)	.068 (L)
N ²⁸	.40 (L)	.82 (L)	.25 (L)
A ⁴⁰	.73 (L)	1.2 (L)	.26 (L)
A ⁴⁰	.38 (M)	.78 (M)	.29 (M)
Kr ⁸⁴	.74 (M)	1.2 (M)	.35 (M)

a (Extrapolated)

It is seen that CCIS sensitivities exceed those of the hot cathode source over the range of parameters investigated, even though the hot cathode source was operated at a relatively high emission current. The sensitivity advantage increases with atomic number; under low mass range operating conditions, the CCIS/Hot Filament sensitivity ratio increases from 1.5 to 4.6 over the mass range 4 amu to 40 amu. Absolute sensitivities range from a low of .064 ma/Torr for helium to a high of 1.2 ma/Torr for krypton and argon. The maximum sensitivity variation with pressure for any particular gas is a factor of two over the approximately two decades investigated.

The sensitivity variation with mass is caused by variations both in the ion transmission of the quadrupole and in the ionization efficiency of the source. The quadrupole manufacturer supplied electronic means for adjusting relative mass sensitivities but these controls were not investigated.

3.10.2 Resolution vs. Atomic Mass - Some variation in resolution as a function of mass is observable (See Figures 11 and 12). This is attributed primarily to arbitrary settings of the manufacturers electronic sensitivity control noted above, since sensitivity can be increased only at the expense of resolution.

4. DISCUSSION

The above results demonstrate the capability of a quadrupole spectrometer to produce spectra with high sensitivity and resolution when coupled to a CCIS. However, the quadrupole used was selected for its commercial availability and does not represent an optimum design for a CCIS. An optimum design is discussed in two previous reports^{10,11}; a larger rf potential and larger physical dimensions (primarily r_0) are required to accommodate the larger ion energy spread and larger ion beam diameter emergent from the CCIS.

An illustration of the benefits of a larger rf operating potential is the improvement resultant from a switch to the low mass range from the medium mass range, described in Section 3.6; it was noted (see Figure 8) that sensitivity increased almost a factor of two and that resolution improved from a 10% valley peak separation of 2.5 amu to 1.5 amu (later 1.0 amu and .7 amu, respectively).

One reason for this improvement can be seen from relationships derived by Paul¹². The rf potential V , mass M , rf frequency ν and the radius of the circle inscribed by the quadrupole rods r_0 , are related by

$$V = \frac{M \nu^2 r_0^2}{2.3 \times 10^{-20}} \quad (4.1)$$

(the constant derives from the use of MKS units). Low mass range resolution and sensitivity improvement exists because the Ultek quadrupole employs higher frequency (5.3 mc) on the low mass range than on the medium mass range (3.3 mc). It can be seen from equation 4.1 that as a result, the rf potential V on the low mass range must be increased to scan the same mass numbers common to both ranges (10-50 amu). However, the larger V required for each mass M increases the transverse ion energy, U_{Tmax} , allowable for 100% transmission, according to

$$U_{Tmax} = V \Delta M / 15M \quad (4.2)$$

where ΔM is the peak width at its base. More ions are, therefore acceptable and sensitivity and/or resolution is improved.

Sensitivity and/or resolution improvement reasonably can be expected to continue by further increases in the required rf operating potential, until the ions with the largest U_{Tmax} are accommodated.

Indicative of higher sensitivity capabilities with larger rf potentials are results obtained with a family of nitrogen spectra recorded as a function of resolution. Here $\Delta M/M$ was varied, instead of V , to reduce the restrictions on U_{Tmax} , according to equation 4.2. Spectra were recorded as in Section 3.1, but subsequent to achievement of otherwise optimum conditions (except for the 425°C bake). Results for the low mass range are shown in Figure 13. It can be seen that when resolution is degraded just short of appreciable peak overlap, N^{28} sensitivity increased a factor of more than 10. On the medium mass range it increased a factor of 15. The .82 ma/Torr sensitivity for nitrogen at .7 amu peak separation (with 10% valley) therefore might reasonably be expected to approach 10 ma/Torr with equal or better resolution by utilizing higher rf potentials; increasing quadrupole dimensions, primarily r_0 , would also assist sensitivity improvement.

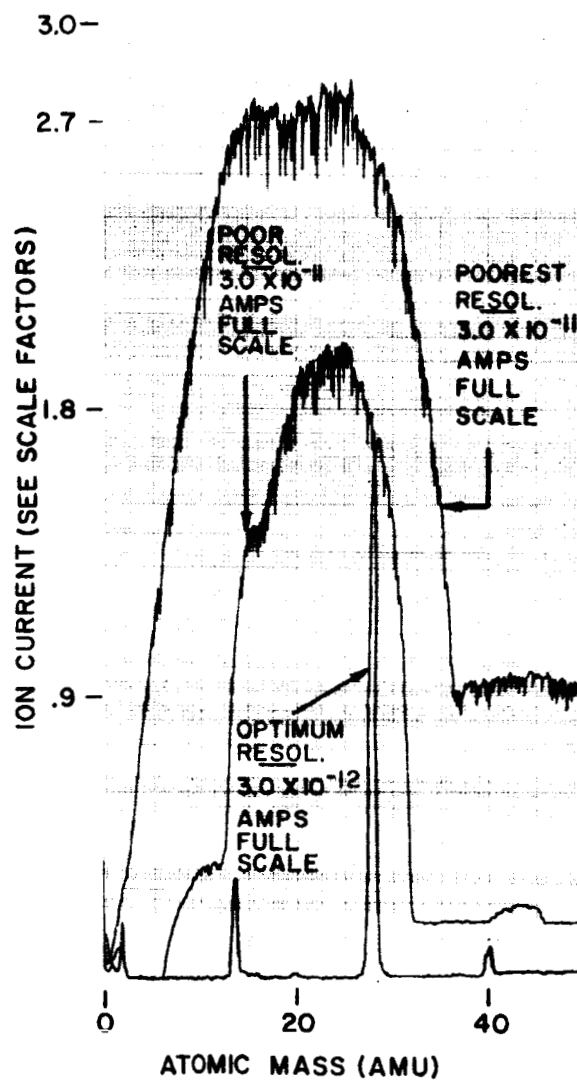


Figure 13

Sensitivity Vs. Resolution-Low Mass Range

If improved quadrupole design allows enlarging the source exit aperture, a still greater fraction of the source's internal sensitivity could be realized; the ion current at the source cathodes K₁ and K₂ give sensitivities in the amps/Torr range.

5. RECOMMENDATIONS FOR FUTURE WORK

This report has shown that the CCIS/Quadrupole spectrometer is a workable instrument for the analysis of residual gases in UHV systems. Some future work is indicated, however, to evaluate its performance more completely and to investigate further improvements. Accordingly, any future work should emphasize the following.

5.1 A direct, simultaneous comparison with a similar hot-filament quadrupole in a typical UHV system. Comparison should investigate sensitivity, resolution, cleanliness, linearity, and ease of operation.

5.2 Investigate response of instrument to single gas species below 10^{-10} Torr. This work should attempt to define if partial pressure sensitivity is independent of total ion source pressure below 10^{-10} Torr.

5.3 Investigate S/N ratio improvements using an electron multiplier.

6. PUBLICATIONS

This work has resulted in a technical paper published with the Contracting Agency cognizance. The reference for this publication is:

Torney, F. L. Jr., and Blum, P.: A Cold Cathode Quadrupole Mass Spectrometer. Fourteenth Annual Conference on Mass Spectrometry and Allied Topics, Dallas, Texas, May, 1966. (ASTM, E-14 Committee) pp. 734-745.

APPENDIX A

SELECTION OF A COMMERCIAL QUADRUPOLE ANALYZER

Careful consideration was given to the selection of a quadrupole mass spectrometer. It was found that commercial instruments lacked the optimum characteristics desired for operation with the cold cathode ion source; these characteristics are primarily sensitivity related and are discussed in previous reports.^{13,14} However, it was concluded that considerable contract effort would be required to construct and test a new quadrupole design. It was therefore decided to purchase the most desirable commercial quadrupole spectrometer and to adapt it to the cold cathode ion source; it was also decided that a commercial instrument would have the advantage of permitting a direct comparison between the performance of a spectrometer utilizing a cold cathode source with one utilizing a hot cathode source, since the latter comes as standard equipment.

Salient characteristics of three commercial quadrupole analyzers, derived from brochures and discussions with the manufacturers, are shown in TABLE A-1. All three instruments are deficient in physical size and in magnitude of the rf potential; two of the instruments operate in an undesirable scanning mode. These considerably reduce the potential sensitivity of the CCIS/Quadrupole spectrometer.

In deciding which of the three to purchase, the Atlas design was discounted primarily for practical rather than technical reasons (Table items a and b). Delivery time, at nine months was too long. Parts, if needed, would also be a long time in arriving since the instrument is made in Europe and, for the same reason, servicing would likely be poor.

Of the two remaining units, the Ultek instrument (manufactured by Electronic Associates, Inc.) has superior resolution* (item c);

* Resolution $M/\Delta M$ is defined differently by Ultek and Varian. The former defines ΔM as the mass separation at which the valley between peaks is 10% of the height of either peak; thus, peaks separated by ΔM are clearly resolved. Varian, however, defines ΔM as the full width at half maximum peak height; peaks separated by ΔM are therefore not as clearly resolved. If Varian's ΔM is multiplied by about 1 1/2 then it assumes the same meaning as Ultek. This is what has been done in making the comparisons.

TABLE A-1 COMPARATIVE QUADRUPOLE SPECTROMETER CHARACTERISTICS

	ULTEK (EAI)		VARIAN		ATLAS
a) Delivery	45 days	30 days	9 months		
b) Where made	USA	USA	GERMANY		
c) Resolution: ΔM separation in amu, required to give a valley height 10% that of either adjacent peak.	$\Delta M \leq 1$ (below 500 amu)	auto. $\left\{ \begin{array}{l} \Delta M \leq 1 \text{ } 1/2 \text{ (below 50 amu)} \\ \Delta M \leq 7 \text{ } 1/2 \text{ (below 100 amu)} \end{array} \right.$ man. $\left\{ \begin{array}{l} \Delta M \leq 2/3 \text{ (below 50 amu)} \\ \Delta M \leq 3 \text{ (below 100 amu)} \end{array} \right.$	$\Delta M \leq 1$ (below 100 amu)		
d) Approximate Price (\$) with a Faraday Cup Detector	13,000	8,000	10,500		
e) RF Potential (volts)	1000	500	800		
f) $\left\{ \begin{array}{l} r_o \text{ (mm)} \\ r_o^2 \text{ (mm}^2\text{)} \end{array} \right.$	$\left\{ \begin{array}{l} 2.7 \\ 7.5 \end{array} \right.$	$\left\{ \begin{array}{l} 3.2 \\ 10.2 \end{array} \right.$	$\left\{ \begin{array}{l} 3.4 \\ 11.6 \end{array} \right.$		
g) Acceptable energy range (ev)	5-50	45-150	Not given		
h) $\left\{ \begin{array}{l} \% \text{ Transmission} \\ \text{Sens.}^* \text{ (amp/to-rr)} \\ \text{Resol. } M/\Delta M \text{ (10\% valley)} \end{array} \right.$	$\left\{ \begin{array}{l} \text{Unknown} \\ (10^{-3} - 10^{-4})^{**} \\ 100 \end{array} \right.$	$\left\{ \begin{array}{l} 100 \\ 2 \times 10^{-3} \\ \text{Zero} \end{array} \right. \left \begin{array}{l} 10 \\ 2 \times 10^{-4} \\ 35 \end{array} \right \left\{ \begin{array}{l} 1 \\ 2 \times 10^{-5} \\ 65 \end{array} \right.$	$\left\{ \begin{array}{l} 100 \\ 2 \times 10^{-4} \\ 35 \end{array} \right. \left \begin{array}{l} 10 \\ 2 \times 10^{-5} \\ 100 \end{array} \right.$		
i) Mode: automatic manual	$\Delta M = \text{constant}$ $\left\{ \begin{array}{l} M/\Delta M = \text{constant} \\ \text{or } \Delta M = \text{constant} \end{array} \right.$	$M/\Delta M = \text{constant}$ $\left\{ \begin{array}{l} \Delta M = \text{constant} \\ \text{or } M/\Delta M = \text{constant} \end{array} \right.$	$M/\Delta M = \text{constant}$ $\left\{ \begin{array}{l} \Delta M = \text{constant} \\ \text{or } M/\Delta M = \text{constant} \end{array} \right.$		

* Using a Faraday cup detector.

** Deduced from manufacturer's specifications for an electron multiplier detector by dividing by a gain of 10^5 and 10^6 (left to right), respectively.

mass peaks of adjacent amu can be resolved to mass 500. In the Varian unit, adjacent mass peaks can be resolved only to about mass 35 in the automatic scan position and to mass 50 when scanning manually; at 100 amu only mass peaks 3 amu apart can be resolved, even when scanning manually (i.e., $\Delta M = 3$ amu); with automatic scanning, ΔM is $7 \frac{1}{2}$ amu at mass 100. As a result, only Ultek meets fully the resolution design goals which have been set, namely resolution of adjacent amu peaks to mass 100 such that the valley between peaks is less than 10% the height of either adjacent peak.

Ultek's greater price (item d) can be attributed to the greater dimensional and electronic tolerances required for its higher resolution and to its high rf potential and consequent increased power requirements.

It was difficult to project which unit would have the best sensitivity when coupled to the CCIS. Ultek has twice Varian's maximum rf voltage (item e) which means it can accept twice the radial ion energy of the Varian unit with consequent increased sensitivity. However, Varian has longer rods and therefore will accept three times the axial ion energy of the Ultek unit (indicated by the higher energy values of item g in the table). Varian's somewhat larger cross-sectional size (indicated by item f), where r_0 is the radius of the circle inscribed by the four quadrupole rods, means that the Varian unit will accept a somewhat larger beam diameter (in proportion to the ratio of r_0^2). If the energy and cross-sectional area were the only factors, Varian would have somewhat of a sensitivity advantage. However, the lower energy limit indicated by the manufacturers (item g) raises an additional point.

A lower energy limit is not discussed in quadrupole theory. (In theory, low ion energy results in high sensitivity.) The lower limit appears to exist primarily because of fringing fields at the quadrupole entrance and exit. It is difficult to see why this effect should differ so significantly between the two units and in particular why the lower limit (45 ev) should be so high in the Varian unit. Inadequate answers were received on the exact meaning of these figures; if accepted as stated, they indicate Ultek to have the sensitivity advantage. This is because the CCIS ions predominate at lower energies (as indicated in Figure 10 of the second final report¹⁵) and because those ions with sufficient energy to overcome a 45 ev limit would be largely discarded by Varian's small radial energy acceptance limit. Unfortunately, answers to

relative sensitivity questions can only be conjectural and could be resolved only after comparative experimental study*.

Another major point of consideration was the scanning mode (item i) whose relative advantages were fully discussed in a previous report¹⁶. From a read-out point of view, there are two types of mass scanning; in one, constant $M/\Delta M$ is maintained; in the other, constant ΔM is maintained. The latter is preferable for the following reasons:

- a) Sensitivity is higher because ion energy restrictions are less stringent.
- b) Sensitivity vs. ion energy is constant with mass.
- c) Mass peaks are equally resolvable over the entire mass range.

(The $M/\Delta M$ mode is by definition called the constant resolution mode; this is misleading, however, since peaks of adjacent amu of low mass can be more easily resolved than those of high mass.) Ultek scans automatically in the desirable, constant ΔM mode; Varian scans in the constant $M/\Delta M$ mode, although it can be made to scan in the desirable mode manually.

In summary, Ultek was indicated to be the preferred spectrometer as to both resolution and scanning mode, with uncertainty existing as to whether it would have a sensitivity advantage over the Varian instrument. The latter question was unresolvable without comparative experimentation. Based on these conclusions, the Ultek spectrometer was purchased.

* Item h in the table shows relative sensitivities, at selected values of resolution, with the manufacturer's own hot filament sources (without the amplification of an electron multiplier tube). These give approximate indications, at best, of relative values that could be expected with the CCIS; for similar resolution, Ultek shows the best sensitivity and Varian the poorest. The percent transmission of ions would be a more useful figure, but it was known only by the Atlas and Varian manufacturers; the former gives a somewhat higher value.

APPENDIX B

QUADRUPOLE PERFORMANCE UTILIZING THE HOT FILAMENT SOURCE

The Ultek/EAI Model 200 Quadrupole* was operated in accordance with instructions supplied by the manufacturer. The primary objective was to establish performance comparisons between the hot cathode and cold cathode ion sources using the same quadrupole analyzer. The following characteristics were examined using the hot cathode source:

- (a) Sensitivity vs. atomic mass number.
- (b) Linearity vs. partial pressure.
- (c) Resolution vs. mass number from 1 to 150 amu.
- (d) Spectrometer cleanliness, noise, and response time.

In Appendix A, it is noted that the Ultek/EAI spectrometer automatically scans the mass spectrum in the most favorable mode (constant ΔM) for the cold cathode ion source (CCIS). It is also important to note that the entrance aperture is nearly equal to the dimension r_0 . Figure 3 of the first quarterly report ¹⁷ shows that this larger aperture can lead to a pronounced variation in transmission as a function of mass number. This variation would lead to exaggerated peak heights for the lighter gases.

One of the main purposes of testing the spectrometer was, therefore, to discover how the quadrupole transmission varied over the desired mass range (1-150 amu). Closely related to this question is the absolute sensitivity of the spectrometer, since the product of the ion source sensitivity and quadrupole transmission factor determines the over-all sensitivity.

* Manufactured by Electronic Associates, Inc., Palo Alto, California and sold by Ultek, Corporation of Palo Alto, California.

Since the sensitivity and resolution are inversely related in any spectrometer, it was first necessary to set the spectrometer to a prescribed resolution and to maintain these conditions throughout the test program. For this purpose, the isotopes of Xenon are nearly ideal. Xe^{131} and Xe^{132} are one mass unit apart and of nearly the same abundance. Therefore, the resolution was set so that the valley between these peaks was 10% of the height of the Xe^{132} peak. This now defines and fixes the resolution for all subsequent tests of the hot filament quadrupole.

The Ultek/EAI spectrometer has three mass ranges:

Low:	1 - 50 amu
Medium:	10 - 150 amu
High:	50 - 500 amu

The performance has not been studied on the 50 - 500 amu range. However, both the "Low" and "Medium" ranges were studied in detail. Before describing the results of these tests, a brief description of the test methods will be given.

The spectrometer was allowed to warm up for 2-3 hours before the start of each test. The electron emission current was set to 3×10^{-3} amperes (recommended value) and the various dials peaked for maximum response for mass 28 (CO^+ , N_2^+). The Modulated Bayard-Alpert gauge was operating and was set to the correct emission for the gas being tested. These emission values were derived as follows:

$$i_A^- = i_N^- \times \frac{S_n}{S_a} \quad (\text{B2.1})$$

where i_A^- is the correct emission current for gas "A" and i_N^- is the correct emission for N_2 (0.3 ma for MBAG sensitivity of .01 amps/Torr). S_n/S_a is the ratio of gauge sensitivity for nitrogen and gas "A" respectively. This ratio was derived from Dushman & Lafferty¹⁸ by normalizing the sensitivities to nitrogen. After appropriate system bake-out, gauge outgassing, and subsequent pumpdown, the test gas was admitted. The ion pump located in the upper section was valved off during the gas admission. The spectrometer was carefully tuned to the appropriate mass peak. The following gases were admitted in the order listed: He, Ne, N_2 , Ar, Kr and Xe.

The system was permitted a 24-hour pumping period between exposures to each gas. The residual system pressure was between $1-2 \times 10^{-9}$ Torr (N_2) before each gas was admitted. An X-Y recorder was used to plot the observed ion current at the selected mass as a function of the Modulated Bayard-Alpert Gauge total pressure of that gas. Pressures up to 10^{-6} Torr of the test gas were used. Data at higher pressures was not taken because the program emphasis is on UHV measurements.

Using the method described, it is possible to determine both linearity and sensitivity for the spectrometer, over a limited range. This was done for both the "Low" and "Medium" mass ranges. For the isotopic gases, suitable corrections were made for the isotope abundances. Table B-1 summarizes this data.

The X-Y recorder traces displayed a very linear relationship between resolved ion current and pressure for He, Ne, N_2 and Argon. For Kr and Xe, a small amount of non-linearity was noted. This problem was also observed during previous tests wherein the UHV ion pump was not valved off. In this instance, the non-linear response was much more pronounced. Although not proven, it is believed that desorption (within the ion pump) of previously pumped gases by the calibration gas produced the observed increase in the Bayard-Alpert gauge response. A similar, but smaller problem may have occurred within the Bayard-Alpert gauge.

TABLE B-1

Sensitivities of Quadrupole 200 for Various Gases

Gas (Mass No.)	Sens. (Low Range) (Amps/Torr) at 3 ma	Sens. (Medium Range) (Amps/Torr) at 3 ma
He ⁴	0.42×10^{-4}	0.178×10^{-4}
Ne ²⁰	0.68×10^{-4}	0.45×10^{-4}
N ²⁸	2.50×10^{-4}	1.98×10^{-4}
Ar ⁴⁰	2.63×10^{-4}	2.86×10^{-4}
Kr ⁸⁴		3.52×10^{-4}
Xe ¹³²		1.42×10^{-4}

TABLE B-1 displays some interesting results. Note that the overall spectrometer sensitivity increases markedly as the mass number increases, and for the "Medium" mass range decreases again at Mass 132 (Xe). It would be anticipated that the ion source sensitivity would increase monotonically with increasing mass due to the increasing ionization efficiency of the gases. It would also be inferred from Figure 3 of the previously mentioned quarterly report that the transmission efficiency of the quadrupole will fall sharply with increasing mass number, particularly for this instrument, in which the r_o^2/d^2 value is approximately unity.

Qualitatively, it would be anticipated that the overall sensitivity of the spectrometer might display a maximum sensitivity at some intermediate mass number. This is because the overall sensitivity will be proportional to the product of the ionizer sensitivity and the transmission factor.

Using the data given in TABLE B-1, it is possible, with certain assumptions, to derive relative transmission factors normalized with respect to nitrogen. For this calculation, the following relation is used:

$$\frac{S_o}{S_n} = \frac{S_a}{S_n} \sigma_a T_r \quad (B2.2)$$

In this equation, S_a/S_n is the sensitivity of the ionizer for gas "A" relative to nitrogen. σ_a is the abundance fraction for the mass being resolved. S_o/S_n is the overall sensitivity of the spectrometer for the mass peak being resolved relative to nitrogen. T_r is the transmission factor for the particular mass relative to the nitrogen transmission.

S_a/S_n values are derived from Dushman & Lafferty by normalizing their published sensitivity factors for nitrogen. The assumption is made that the hot filament ionizer will exhibit the same sensitivity factors. This assumption is reasonable since the electron energies used in the quadrupole ionizer are nearly equal to those used in Bayard-Alpert gauges (90 volts).

Rearranging equation (B2.2),

$$T_r = \frac{S_o}{S_n} \times \frac{S_n}{S_a} \sigma_a \quad (B2.3)$$

TABLE B-2 below shows how the relative transmission, calculated from the data of TABLE B-1 varies with mass number.

TABLE B-2

Quadrupole Transmission Vs. Mass						
(normalized to nitrogen)						
(Mass No.)	ϕ_a	$\frac{S_o/S_n}{\text{(Low)}}$	$\frac{S_o/S_n}{\text{(Med)}}$	S_n/S_a	ϕ_a	$T_r \text{ (Low)}$ $T_r \text{ (Med)}$
He ⁴	1.00	0.168	0.090	6.45		1.08 0.58
Ne ²⁰	0.905	0.272	0.227	4.64		1.26 1.07
N ₂ ²⁸	1.00	1.00	1.00	1.00		1.00 1.00
Ar ⁴⁰	1.00	1.05	1.45	0.84		0.883 1.22
Kr ⁸⁴	0.569	--	1.78	0.95		-- 1.69
Xe ¹³²	0.269	--	0.723	1.36		-- 0.982

Using equation (B2.3), the transmission factors (T_r) have been calculated and normalized with respect to nitrogen. These factors are shown in the last two columns of this table, for both the "Low" and "Medium" mass ranges of the instrument. It is apparent from these data that the quadrupole transmission is not varying with mass number in the manner predicted by the theory. In fact, it is remarkably constant, at least for the "Low" mass range.

The manufacturer (EAI) was questioned regarding this unexpected variation of transmission vs. mass number. In a private communication, it was disclosed that certain bias voltages have been provided in the electronics which supply the d-c quadrupole potentials. These small potentials are provided to compensate for increased quadrupole transmission at the low mass numbers only.

Compensation is accomplished by essentially improving the resolution at the expense of lowered transmission. Therefore, the transmission for the lower masses can be adjusted to be more nearly equal to the transmission at the higher masses. A noticeable reduction in peak width, ΔM , should coincide with this reduction in the transmission factor.

Figures 14 and 15 show complete spectra of a mixture of inert gases taken on the "Low" (1-50 amu) and "Medium" (10-150 amu) mass ranges of the hot filament quadrupole. A careful examination of Figure 15 reveals some interesting qualitative information. Xenon 131 and 132 have been clearly resolved with a valley between these peaks of approximately 10%. However, Krypton 82 and 83 have not been as well resolved. At the low end of the mass scale, the Hydrogen and Helium peaks are noticeably more narrow than those at any other part of the entire mass range. If these results are correlated with the TABLE B-2 transmission factors (Medium range), we see the anticipated inverse relationship between resolution and transmission. Similarly, a comparison can be made between TABLE B-2 transmission factors and Figure 14. As both Figure 14 and TABLE B-2 show, however, the variation in resolution is less for the "Low" mass range than for the "Medium" range.

Thus far, the absolute values of the quadrupole transmission have not been discussed. It is of importance to know what these absolute relations are, since transmission is closely related to the spectrometer sensitivity. Furthermore, absolute data should be available in order to compare the hot and cold cathode ionizers. The absolute transmission factor is difficult to determine, however, since the ion beam which enters the aperture hole cannot be measured directly. This beam is not perfectly collimated and therefore some ions will be lost on the rods and other structures even though the quadrupole voltages are off. Under these circumstances, it is necessary to measure the ion current at the specified resolution and at zero resolution. The transmission factor is then the resolved current divided by the unresolved current. In making this measurement, it is important to measure only one gas at a time. Also, appropriate corrections must be made for the abundance of the isotope being resolved.

The data shown in TABLE B-3 below was obtained using this method of determining absolute transmission.

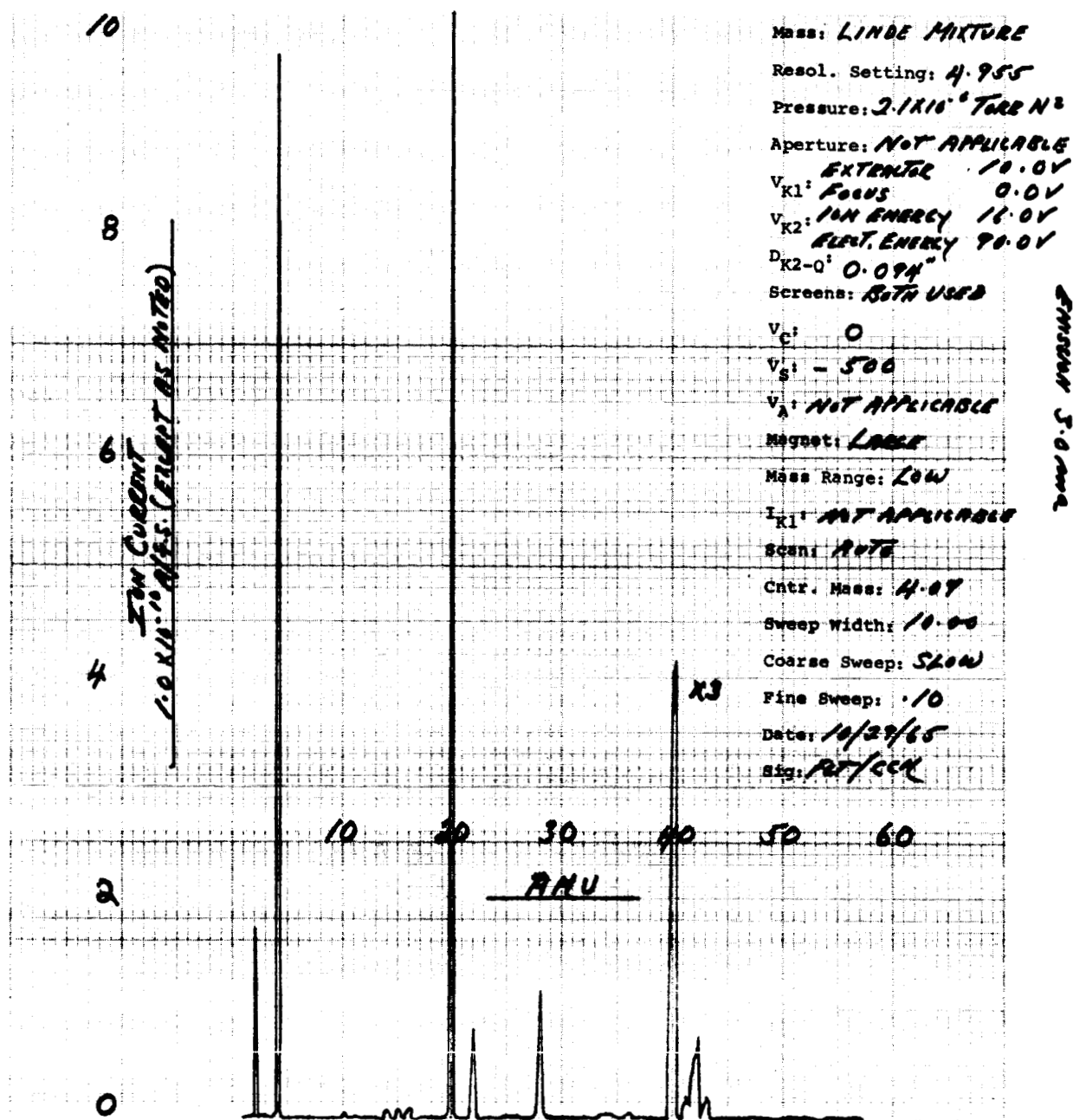


Figure 14
 Low Mass Range Spectrum Taken With The Hot Filament Spectrometer

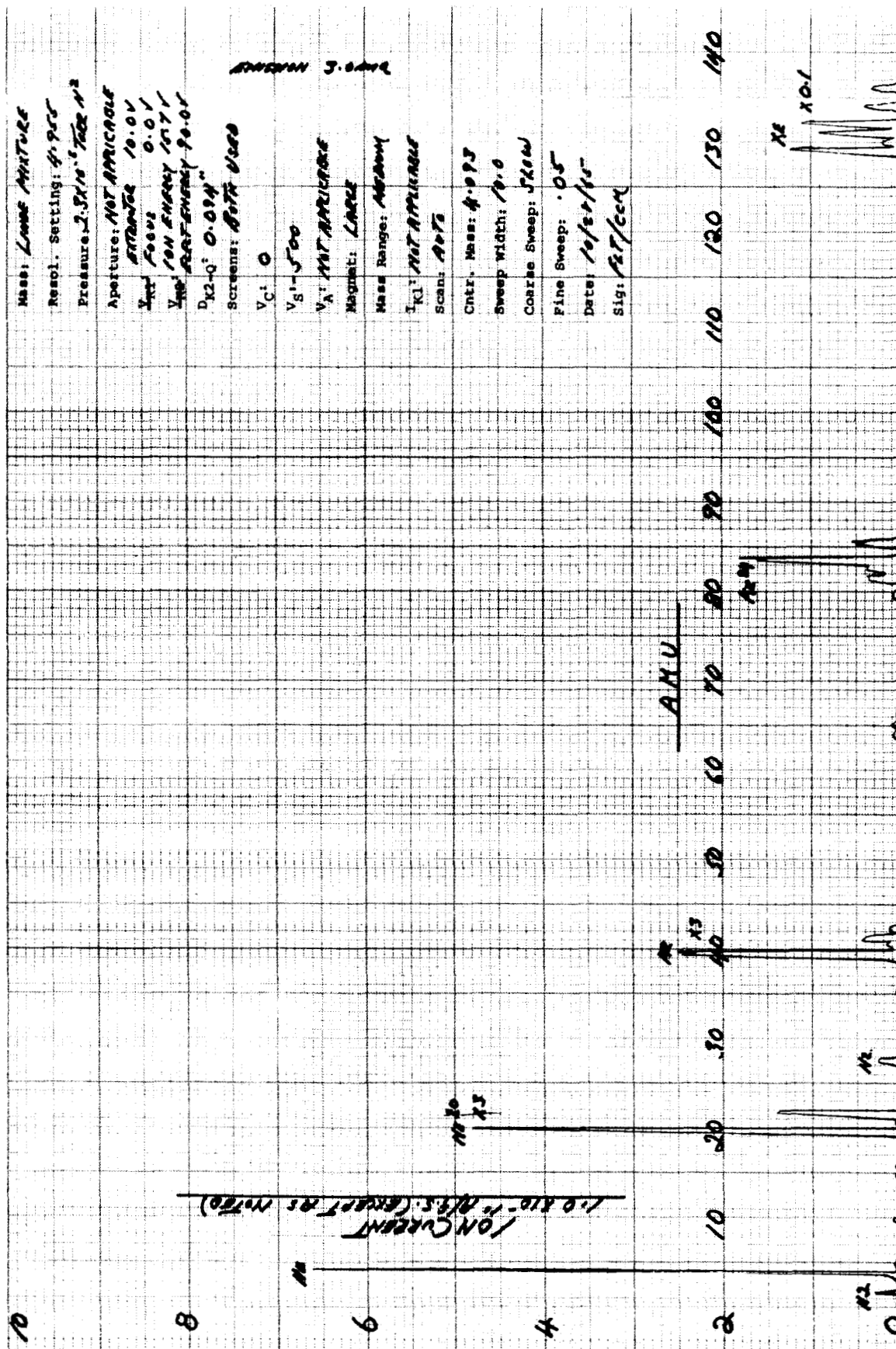


Figure 15
 Medium Mass Range Spectrum Taken With The Hot Filament Spectrometer

TABLE B-3

Absolute Transmission Vs. Mass Number

Gas (Mass No.)	σ_a	T_a (Low Range)	T_a (Medium Range)
He ⁴	1.00	Not Measured	0.222
Ne ²⁰	0.905	" "	0.283
N ₂ ²⁸	1.00	" "	0.286
Ar ⁴⁰	1.00	" "	0.344
Kr ⁸⁴	0.569	" "	0.435
Xe ¹³²	0.269	" "	0.299

If the data of TABLE B-3 is normalized for T_a (nitrogen), the results are in reasonably close agreement with the values given in the last column of TABLE B-2. Similar absolute transmission factors were not determined for the "Low" mass range because significant changes were not evident in TABLE B-2.

Thus, it is seen that the performance of the hot-filament quadrupole is in agreement with quadrupole theory and with the manufacturer's specifications. In order to derive quantitative results from the instrument, more attention should be given to the proper adjustment of the controls which effect quadrupole transmission and resolution for the lighter gases.

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